

# Calibration of Germanium Resistors at Low Temperatures (2–20° Kelvin)

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Two germanium resistors, C and D, have been referenced to values of temperature determined by the NBS acoustical thermometer. The resistance-temperature calibration data for these resistors have been fitted to a function of the form  $\log_{10} R = \sum_{n=0}^m A_n (\log_{10} T)^n$  and the results are presented. The resistors C and D that are used as secondary standards maintain the scale, NBS Provisional Scale 2–20 (1965), to which public calibrations of germanium thermometers are referenced.

The calibration apparatus and measurement techniques that are employed in calibrating submitted resistors are described in detail. Additionally, data of three typical calibrations and their treatment by polynomial fitting are included to demonstrate the characteristics of some commercially available germanium resistors.

**Key Words:** Calibration, germanium resistors, low temperatures, secondary thermometers, thermometry.

## 1. Introduction

Recently at the National Bureau of Standards a provisional temperature scale in the region of 2 to 20 °K was established, based on temperature values derived with the Acoustical Thermometer [1, 2],<sup>1</sup> a primary thermometer. The temperature scale is presently maintained with six germanium resistance thermometers. Their calibration is in close agreement [2] with other secondary thermometers in regions where temperature calibrations overlap, i.e., 2 to 5 °K, the  $T_{3a}$  He<sup>4</sup> Vapor Pressure Scale [3] and 12 to 20 °K, NBS (1955) Scale [4].<sup>2</sup>

In practice, temperature sensing devices should be independent of thermal history and have a measurable temperature coefficient which covers a prescribed temperature range as a smooth function of some parameter. Some materials, i.e., carbon, [5, 6, 7, 8], metals, [9, 10, 11], and piezoelectrics [12] meet these requirements but are limited in the cryogenic temperature range 2 to 20 °K because of insensitivity, instability, or nonreproducibility. Some of the pure metals become superconducting, i.e., indium [13] and lead at temperatures of 3.4 and 7.2 °K, respectively. Impurity doped germanium semiconductors [14, 15, 16, 17, 18, 19] exhibit a temperature-dependence of electrical resistance such that they are readily adaptable to the needs of precise low temperature thermometers. By controlling the impurity doping of germanium, thermom-

eters can be produced to cover temperatures within various ranges of interest, i.e., 1 to 20 °K, 20 to 50 °K, etc. Within the useful range of these thermometers two conduction processes are involved [20]; at temperatures below 10 °K, impurity conduction is the dominant mechanism, while free hole conduction limits the resistivity at the higher temperatures. To cover the region 2 to 20 °K, a point-by-point calibration is necessary because of the complicated conduction processes; at the present time an accurate simple analytical representation of resistance versus temperature for germanium resistors does not exist.

We will discuss the NBS Provisional Scale 2–20 (1965) and a "Germanium Thermometer Calibrator" which is employed in calibrating germanium thermometers against it.

## 2. NBS Provisional Temperature Scale 2–20 (1965)

The temperature scale, against which calibrations are performed, is called NBS P 2–20 (1965) and is based upon values of absolute temperature that have been determined with the NBS Acoustical Thermometer. This instrument and its operation have recently been described [2] but a more detailed description (including tabular data that defines the isotherms) will appear in the near future.<sup>3</sup>

During the operation of the acoustical thermometer it was essential that secondary thermometers (in this

<sup>1</sup> Figures in brackets indicate the literature references at the end of this paper.

<sup>2</sup> The present NBS temperature scale in this region, 12 °K and above, is maintained with platinum resistors, designated NBS (1955), and defined to be 0.01 deg lower than the scale given in reference [4].

<sup>3</sup> To be published in a future issue of *Metrologia*.

instance, germanium resistors that were in intimate thermal contact with the acoustical thermometer were used) be employed to indicate the constancy of maintaining an isotherm temperature,<sup>4</sup> as well as to retain a calibration of the acoustically determined isotherm temperature. This is accomplished by periodically monitoring the resistance values of the germanium resistors while the acoustical thermometer is in operation. Two particular germanium resistors, which we can label A and B, have always served this function and thus they have been calibrated by the acoustical thermometer. Additionally, two other germanium resistors have been mounted in the apparatus and have been used as additional monitoring resistors in the acoustical thermometer. We shall name two such resistors C and D. While C and D have been used for monitoring the acoustical thermometer for certain specific isotherms, they give reference to all of the isotherms through calibrations against resistors A and B.

Quite naturally the calibrations of C and D against A and B did not always take place at exactly the original acoustically-determined isotherm temperatures and thus a knowledge of  $\frac{dR}{dT}$  (for resistors A and B) was required for properly assigning temperatures to C and D. Because the calibration temperatures of C and D were never more than 0.035 °K from the acoustically determined isotherms, the required interpolations and knowledge of  $\frac{dR}{dT}$  (for A and B) did not critically affect the calibration of resistors C and D ( $\frac{dR}{dT}$  values could be established with sufficient accuracy for this interpolation from the calibrations of A and B). In fact the temperature of only six calibration points for resistor C differed more than 0.020 °K from the original acoustically defined isotherm-temperatures; for D, one was 0.016 °K and the remainder less than 0.012 °K. The resistors C and D were never in the acoustical thermometer together so their calibrations were performed independently with regard to time and environmental location within the acoustical thermometer apparatus.

At this point it is necessary to note that previous to their calibration some laboratory history of resistors C and D had been accumulated; this has been reported in an earlier article [21]. The resistors were repetitively, thermally cycled between room temperature and 4.2 °K, with systematic resistance-temperature calibrations performed at a temperature close to 4.2 °K. A "constant temperature" liquid helium bath contained the resistors during the 4.2 °K portion of the cycle and thus, by measurements of the liquid helium vapor pressure, a very reproducible temperature reference (to the  $T_{28}$  scale) was obtained. Resistor C had been thermally cycled 88 times and demonstrated a reproducibility of the 4.2 °K calibration within 0.0011 °K; resistor D underwent 86 cycles and demon-

strated a constancy within 0.0005 °K. The estimated error from various measurements in the cycling process was equivalent to 0.0004 °K. Appropriate attention has always been applied to the measuring currents through the resistors to avoid undesired, appreciable Joule heating.

After being calibrated with reference to the acoustical thermometer, the resistors C and D were placed within the calibrator, which will be described in this article, and measurements of their resistances were conducted at temperatures which closely approximated the original isotherm temperatures. More specifically, the temperature of the apparatus was controlled until the resistance measurement of one resistor, for example, C, indicated that approximation to an original acoustical isotherm temperature had been achieved. Next, at this temperature a value was obtained for the resistance of D. Thus there was afforded a comparison of indicated temperatures between C and D within the calibrator since, to repeat, each resistor had been previously independently calibrated with reference to the acoustical thermometer. Temperature discrepancies between C and D (after appropriate small interpolations had been made), from comparison measurements within the calibrator, never exceeded 0.003 °K. Consequently our assignment of temperature values to future calibrations in the calibrator against C and D is slightly arbitrary. The maximum temperature discrepancy of 0.003 °K between resistors C and D, as obtained during the calibrator use, probably indicates that the combined errors of electrical measurements and temperature controlling correspond to about  $\pm 0.002$  °K.

Thus temperatures have been associated with resistors C and D for their use as secondary standards in the calibrator. This information is presented in columns 1 and 2 of tables 1 and 2. We were desirous of possessing more information about the resistors' calibrations and consequently have performed a computer analysis of the data. A polynomial of the form

$$\log_{10} R = \sum_{n=0}^m A_n (\log_{10} T)^n$$

was fitted to the  $R_{\text{data}}$  and  $T_{\text{data}}$  calibration data of each resistor by the method of least squares. A double precision computer program prepared by the Statistical Engineering Laboratory was used to overcome the roundoff error characteristic of fitting high order polynomials, and in this case the roundoff error was several orders of magnitude smaller than other experimental uncertainties. All data were given equal weight and  $m$ , the degree of the polynomial, was varied from 3 to 8. The eighth order polynomial produced a fit to the input data such that no datum point deviated from the polynomial by more than 0.0045 °K. The reduction in the sum of squares of deviations due to the addition of terms of higher order was statistically significant (0.05-probability level) in all cases up to and including the 8th degree. We have not extended the analysis since we feel that fit-

<sup>4</sup> An isotherm as determined by the acoustical thermometer consists of the speed of sound in helium gas as a function of the gas pressure.

ting the data within 0.004 °K is a reasonable limit. It is noteworthy to mention that as the lower temperature data points are excluded, comparable fitting is possible with fewer terms in the polynomial.

After obtaining values of the 9 coefficients we effected a computer generation of temperature, resistance,  $\frac{dR}{dT}$ ,  $\frac{d^2R}{dT^2}$ , and  $\frac{1}{R} \frac{dR}{dT}$  at intervals of 0.010 °K.

Using the generated  $\frac{dR}{dT}$ , simple interpolations have been made to obtain values of generated resistance,  $R_{gen}$ , which correspond to the input temperatures,

TABLE 1. Resistor C

$T_{data}$ <sup>a</sup>	$R_{data}$ <sup>a</sup>	$R_{gen}$ <sup>b</sup>	$R_{data} - R_{gen}$	$\frac{dR}{dT}$ <sup>c</sup> (Ohms/0.001 °K)	$\Delta T$ <sup>d</sup> (0.001 °K)
°K	Ohms	Ohms	Ohms		
2.321	11.481	11.4814	-0.4	-13.5	0.0
2.805	7.056.7	7.056.7	2.0	-6.12	-3
3.207	5.122.5	5.125.0	-2.5	-3.71	-7
4.206	2.800.9	2.798.9	2.0	-4.43	-1.4
5.020	1.916.0	1.917.8	-1.8	-0.817	2.2
6.060	1.272.9	1.271.8	1.1	-4.51	-2.3
6.977	918.51	919.16	-0.65	-3.12	2.1
8.066	645.32	645.29	.03	-3.00	-1.5
8.989	489.94	489.93	.06	-3.40	-0.4
9.889	362.65	362.53	.12	-3.10	-1.2
10.901	296.86	296.82	.04	-0.70	-0.6
12.018	230.85	230.97	-0.12	-0.99	2.4
12.962	190.74	190.82	-0.08	-0.57	2.2
14.036	157.05	157.01	.04	-0.27	-1.5
14.993	134.26	134.21	.05	-0.21	-1.0
16.050	114.77	114.76	.01	-0.16	-0.6
16.970	101.36	101.32	.04	-0.13	-3.1
18.004	89.051	89.100	-0.049	-0.11	4.5
18.940	79.497	80.006	-0.009	-0.008	1.0
20.054	71.094				

<sup>a</sup>  $T_{data}$  and  $R_{data}$  refer to calibration quantities, temperature and resistance.  
<sup>b</sup>  $R_{gen}$  is the resistance, at the temperature  $T_{data}$ , obtained by generating resistance as a function of temperature from the evaluated polynomial coefficients  $A_n$ .  
<sup>c</sup>  $\frac{dR}{dT}$  is generated from the evaluated coefficients  $A_n$ .  
<sup>d</sup>  $\Delta T$  is obtained by dividing  $|R_{data} - R_{gen}|$  by  $\frac{dR}{dT}$ .

TABLE 2. Resistor D

$T_{data}$ <sup>a</sup>	$R_{data}$ <sup>a</sup>	$R_{gen}$ <sup>b</sup>	$R_{data} - R_{gen}$	$\frac{dR}{dT}$ <sup>c</sup> (Ohms/0.001 °K)	$\Delta T$ <sup>d</sup> (0.001 °K)
°K	Ohms	Ohms	Ohms		
2.321	10.847	10.848	-1.0	-12.8	9.1
2.805	6.666.1	6.653.2	2.9	-5.8	-0.5
3.207	4.820.3	4.824.2	-3.9	-3.5	1.1
4.206	2.630.3	2.637.0	-3.3	-1.4	-2.4
5.020	1.792.5	1.795.1	-2.6	-0.77	3.1
6.060	1.186.3	1.185.4	0.9	-0.44	-2.5
6.977	852.60	853.10	-0.50	-0.29	1.7
8.066	596.26	596.39	-0.13	-0.19	-0.4
8.989	451.44	451.34	0.10	-0.13	-0.8
9.889	351.62	351.60	.02	-0.07	-3
10.901	272.27	272.32	-0.05	-0.05	.4
12.018	211.33	211.55	-0.22	-0.043	.4
12.962	174.68	174.77	-0.09	-0.04	2.6
14.036	143.80	143.78	.02	-0.025	-0.8
14.993	122.98	122.98	0	-0.019	-0
16.050	105.21	105.20	.01	-0.015	-0.7
16.970	92.96	92.94	.02	-0.012	-1.7
18.004	81.743	81.785	-0.042	-0.0097	4.3
18.940	73.469	73.494	-0.025	-0.0080	3.1
20.054	65.351	65.341	0.010	-0.0066	-1.5

<sup>a</sup>  $T_{data}$  and  $R_{data}$  refer to calibration quantities, temperature and resistance.  
<sup>b</sup>  $R_{gen}$  is the resistance, at the temperature  $T_{data}$ , obtained by generating resistance as a function of temperature from the evaluated polynomial coefficients  $A_n$ .  
<sup>c</sup>  $\frac{dR}{dT}$  is generated from the evaluated coefficients  $A_n$ .  
<sup>d</sup>  $\Delta T$  is obtained by dividing  $|R_{data} - R_{gen}|$  by  $\frac{dR}{dT}$ .

$T_{data}$ . These resistances,  $R_{gen}$ , are listed in column 3 of tables 1 and 2. Column 4 shows the difference between the input and generated resistance: by again employing the generated  $\frac{dR}{dT}$  of column 5 we obtain the temperature difference ( $\Delta T$  of column 6) which corresponds to the resistance differences of column 4. It is readily apparent from column 6 of tables 1 and 2 that the temperature-resistance data for resistors C and D can be represented by an eighth order polynomial within 0.005 °K.

Additional information is obtained concerning the polynomial and therefore, the resistors also, since (for each resistor) the generated  $\frac{d^2R}{dT^2}$  does not change its sign; the fitted polynomial function is monotonically smooth. Consequently one is reasonably confident that the fitted polynomials can be used for interpolating between original calibration points. Figures 1 and 2 depict the variation of  $R$  versus  $T$ , and  $\frac{1}{R} \frac{dR}{dT}$  versus  $T$ , respectively, for both resistors, C and D.

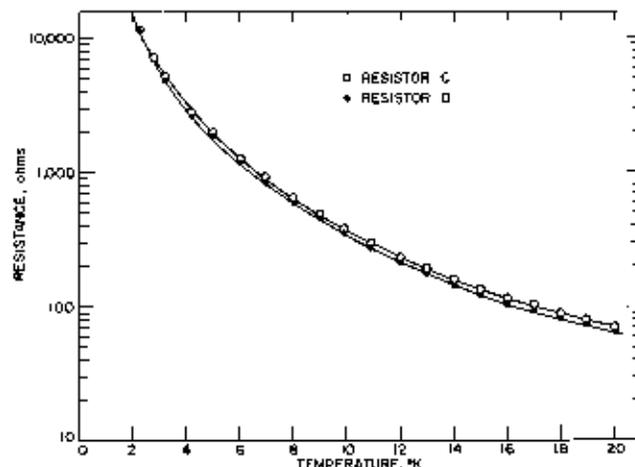


FIGURE 1. A plot of resistance as a function of temperature: □, Resistor C; ●, Resistor D.

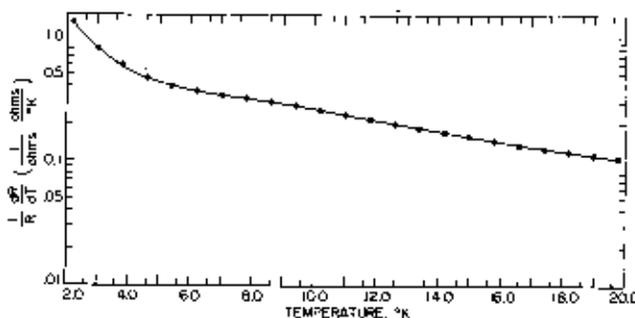


FIGURE 2. Resistance-temperature sensitivity,  $\frac{1}{R} \frac{dR}{dT} \left( \frac{1}{°K} \right)$ , as a function of temperature (°K). The plot is applicable to both C and D since their temperature sensitivities do not differ more than one percent.

While resistors C and D are the basis for present calibrations from 2 to 20 °K at the National Bureau of Standards, several other resistors exist that have been similarly treated; additionally, resistors A and B are still intact within the acoustical thermometer. Resistor D, prior to its mounting in the acoustical thermometer, had been calibrated at the National Bureau of Standards against the NBS (1955) scale below 20 °K. It was one of the two thermometers that afforded a comparison, (which has been reported previously [1, 2]) between values of acoustically determined temperatures and the NBS (1955) scale.

### 3. Calibrator<sup>5</sup>

#### 3.1. General Design Considerations of Calibrator

Central to apparatus which we have constructed is a solid copper cylinder. It is required, during the course of calibration measurements, that the copper mass and its contained resistors be maintained at accurately controllable temperatures at approximately every degree from 2 to 20 °K. To achieve this temperature regulation the system must be quasi-thermally isolated from its surroundings. The copper mass and imbedded thermometers are shown in figure 3. In operation (the smaller can having been soldered in place) the copper mass and small can are included in the large can. If a vacuum is maintained within the larger can, then the small can and its contents are partially isolated, thermally, from the environment that contains the large can.

<sup>5</sup>Certain commercial materials and equipment are identified in this section in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

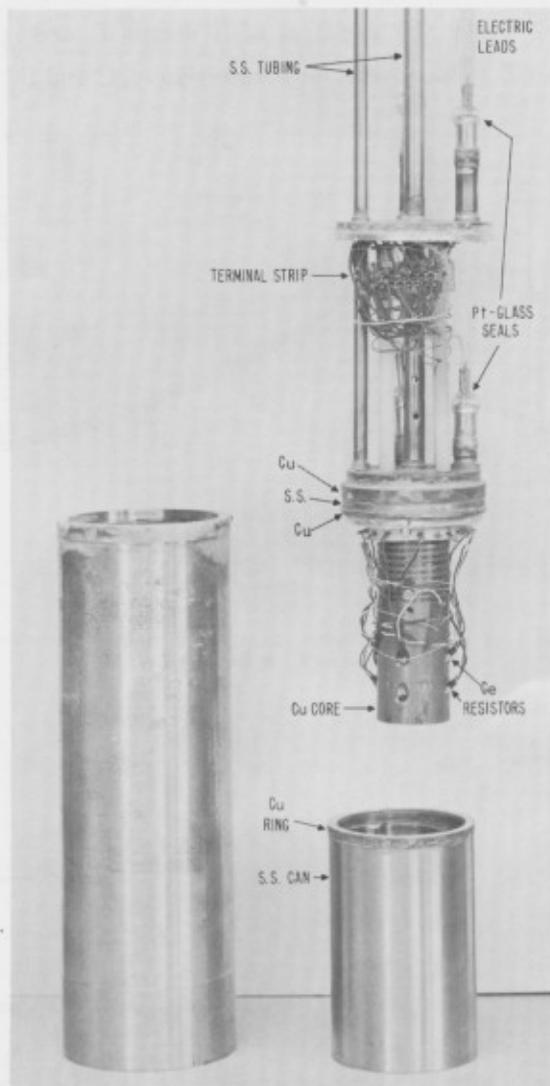


FIGURE 3. Closeup photograph of the calibrator that is used in the temperature calibration of germanium resistors.



FIGURE 4. Assembled calibrator.

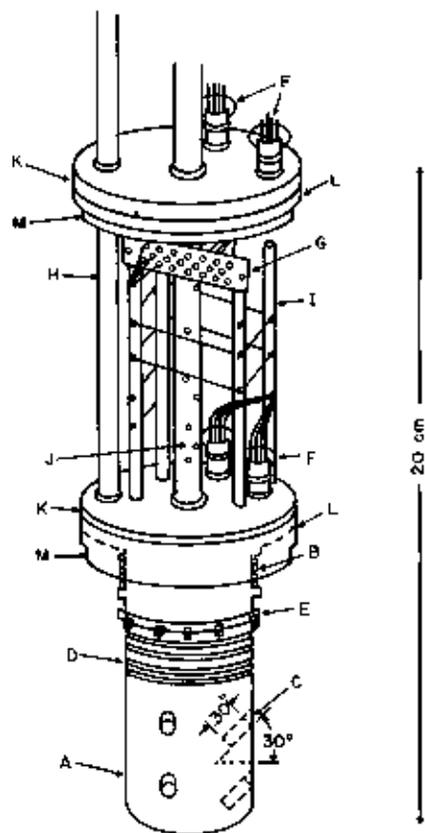
Within the vacuum enclosure heat transfer by gas conduction and convection is essentially eliminated, and the small can will reduce radiant heat flow between the thermometers and the outer vacuum can. There remains, however, a thermal connection between the inner assembly and the outer can via electrical leads and mechanically supporting members, i.e., the two stainless steel tubes. The inner assembly is thermally floated at temperatures that exceed the environmental temperature of the outer can, with a resultant temperature gradient along the electrical lead wires and stainless steel tubes.

A more complete view of the calibrator is depicted in figure 4. Uppermost is the Dewar cap, which affords ingress of electrical leads and the stainless steel tubings. Because the lower portion is in a liquid helium bath during calibrations any heat from ambient temperatures that is conducted downward along the leads and tubings is dissipated in the refrigerant. Thus it is evident that under steady state conditions, the interesting heat flow occurs from the inner assembly toward the top of the outer vacuum can. The dimensions of the supporting tubes and electrical leads have been selected so that accurate temperature control of the copper core and resistors is attained through a reasonable expenditure of electrical heating to the copper core.

### 3.2. Main Copper Core

The main core (A) of the isothermal system in figure 5 was constructed of 58.93 mm (diameter) copper rod. Cavities 3.58 mm (diameter) drilled at 60° angles to its axis serve as receptacles for the germanium thermometers. Also machined in the core was a spool-like section (B) upon which the heater coil was wound. Directly above the coil form is the cap (K) for the inner chamber. Silver soldered to the cap's perimeter is a stainless steel tubing sleeve (L) 0.30 mm thick, 12.70 mm long. Soldered on the lower end of the tubing is a copper ring (M) with a tongued edge to receive the groove of the small vacuum can of figure 3. In the cap (K) two 9.53 mm holes were drilled for the platinum glass seals, a 6.35 mm hole for an evacuation tube (H), a 9.53 mm opening for the supporting tubing (J), and four screw threads were tapped for the nylon rods (I). On the copper core below the coil form a spiral groove (D), 1.27 mm deep, three threads per cm, was routed from the core's perimeter to serve as a channel for thermally anchoring the lead-in wires that return upwards to the terminal ring (E).

The terminal ring was fabricated from Teflon tubing and contains terminal lugs made of 0.76 mm platinum wire. The wire has been flattened on one extremity and cut to extend 9.53 mm beyond the Teflon ring. Holes 0.50 mm were drilled at the end of the platinum lugs to secure the thermometer electrical leads prior to their soldering. Two set screws attached the ring to the copper core.



- |   |                             |   |                          |
|---|-----------------------------|---|--------------------------|
| A | Cu Core                     | G | TERMINAL STRIP           |
| B | HEATER COIL                 | H | PUMPING LINE (INNER CAN) |
| C | CAVITIES FOR THERMOMETERS   | I | NYLON                    |
| D | .05" DIA. GROOVES FOR WIRES | J | OUTER CAN PUMPING LINE   |
| E | ANNULAR TERMINAL STRIP      | K | CU CAPS OF CANS          |
| F | PLATINUM GLASS SEALS        | L | STAINLESS STEEL RING     |
|   |                             | M | CU RING                  |

FIGURE 5. Detailed drawing of the calibrator low-temperature components.

### 3.3. Vacuum Chambers

For thermal isolation the above components were encased within a vacuum tight cannister. The vacuum can was constructed of stainless steel tubing 58.93 mm diam, 114.30 mm long and 0.30 mm thick with one capped end of 0.50 mm thickness. A copper ring was silver soldered to the open end of the can; the ring was grooved to receive the tongued edge (M) of the can top.

The outer can top was also made of copper and holes, similar to those of the inner can, were drilled for the insertion of seals and evacuation lines. There also, a stainless steel sleeve that terminates in a tongued copper ring was soldered to the cap's circumference. The reason for using the stainless steel sleeves in both vacuum systems may not be obvious. In assembling the apparatus, heating is required to solder the

vacuum cans to the flanges (M). If the heat were sufficiently great, electrical lead insulation might be damaged in regions where the insulation is in contact with copper (the spiral groove (D) and the vacuum can tops). Additionally, heating of the large copper core would elevate the temperature of the germanium thermometers to such an extent that they might be damaged. Precautions have been taken to avoid such heating. (a) Wood's metal solder is used for assembly because its melting point is relatively low. (b) The stainless steel paths (L) provide a sufficiently large thermal resistance that the solder joints can be made with only a slightly perceptible temperature rise in the vacuum can tops and the copper mass (A). A similar design has been employed in mounting the platinum glass seals (F) to simplify their replacement.

### 3.4. Electrical Seals and Wiring

Glass-metal type electrical lead-in seals were used exclusively in the construction of the isothermal calibrating system. For ambient conditions, Kovar-glass type capillary-tubing seals were utilized. At temperatures where components are immersed in cryogenic liquids, our experience has shown the necessity for using platinum-glass type seals. The NBS glass shop made the 9-capillary platinum-glass seals<sup>6</sup> that were incorporated in the system.

At ambient temperatures where electrical leads entered the Dewar system, eighteen #38 AWG double nylon, enamel coated copper wires were fed through and soldered to individual capillaries of two 9-capillary Kovar-glass seals that were located on the Dewar cap. The wires continued from the ambient seals, were strung through and soldered to individual capillaries of two platinum-glass seals (F) of the outer can, and terminated at the 20-pin rectangular terminal strip (G).

From the terminal strip, 1.54 m lengths of Evanohm wire were spiraled around the notched nylon supporting rods (I) in six windings and the remaining lengths of wire were passed through and soldered at the individual capillaries of the two platinum-glass seals that are mounted on the top of the inner vacuum can. The remaining lengths of wire within the inner can region were then snugly wound into the spiral groove (D) of the main copper core (A) and terminated at the nylon ring (E). The strands of Evanohm wire were wound around the nylon rods to lengthen the thermal resistance path along the lead wires, in effect, to reduce the temperature gradient along the electrical leads, since the temperature difference between the isothermal system and its heat sink (cryogenic bath) can differ by as much as 18° during a calibration run. The enamel coated Evanohm lead wires were placed in physical contact with the copper core to enhance thermal equilibrium between resistance thermometers, electrical leads and the environment within the calibrating unit. Evanohm wire was used because its

resistance-temperature coefficient is quite low.<sup>7</sup> The heater coil was noninductively wound on spool (B) of figure 5, with 61.5 m of the above wire. The wire diameter is 0.10 mm and the wire has a resistance of 1.65 Ω/cm.

## 4. Germanium Thermometers

### 4.1. General Description

The bridge-shaped encapsulated germanium thermometer, shown in figure 6 with current contacts external to the potential contacts, has advantages not found in some other types. The germanium element is mounted in a relatively strain-free manner within its encapsulation to reduce piezoresistance [20, 22] that is exhibited by germanium at low temperatures. Effects of contact resistances and possible thermal strains in the contacts are relatively unimportant because measurements are made potentiometrically and temporal changes in contacts do not necessarily change the thermometer calibration [20]. Encapsulation of the thermometer element in helium exchange gas aids the germanium element in achieving thermal equilibrium with its metallic capsule.

### 4.2. Mounting of Thermometers

Leads of the germanium thermometers are soldered with Wood's metal to the platinum lugs of the nylon terminal ring (E) using a low-powered soldering iron to minimize overheating of the germanium element through heat conduction. A very dilute solution of Baker's Flux (aqueous solution of the chlorides of Zn and NH<sub>4</sub>) is used and upon completion of the soldering, the terminal junctions are flushed with a swab and hot water to remove traces of flux.

The casings of the thermometers are placed within the copper cavities. A primary consideration in the calibrator design is the attainment of thermal equilibrium between the resistors (the sensor, secondary standards, and those to be calibrated). Since the resistors are generally encapsulated, one must be concerned with the thermal equilibrium between each resistor and its encapsulation, as well as between all

<sup>7</sup> At 20 and 273 °K, its resistivity is 132.5 and 133.3 μΩ cm, respectively [25].

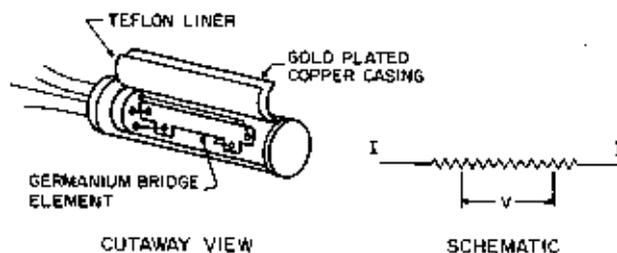


FIGURE 6. Schematic portrayal of a typical germanium resistor encapsulation.

<sup>6</sup>The seals were thermally cycled from ambient to liquid helium temperatures at least three times and leak tested on a mass spectrometer type helium leak detector prior to their use in the calibrator.

encapsulations. After the germanium thermometers are wired into the circuit and continuity and shorting checks made, the inner-most vacuum can is soldered into place with Wood's metal, using a modified soldering gun that possesses an annular copper heater element for uniform heating. Then the outer vacuum can is soldered into place using a gas-air torch of fine orifice. Both vacuum systems are leak detected, and again checks are made to assure circuit continuity and adequate electrical insulation between the d-c electrical circuits and "ground." Insulation resistances of 10 MΩ—the limit of a portable Wheatstone bridge—are commonly measured.

## 5. Temperature Measurements and Control

### 5.1. Refrigerant Reservoir

For temperature-calibration measurements 5 to 20 °K or above, the helium bath into which the calibrator is immersed, is vented to atmospheric pressures. The bath at this temperature provides an adequate heat sink for dissipating the energy emitted from the calibrator. For temperature measurements 2 to 5 °K, the bath pressure is reduced to achieve a bath temperature of 1.8 °K or less. A requirement for temperature stability within the calibrator is that there should be a current flow to the heater coil at all times, and experience has shown that the minimum power output of the heater should be 6.25 μW (25 μA through the 10,000 Ω heater coil), otherwise the required stability and reproducibility of temperatures is not possible.

### 5.2. Temperature Control

Temperature control is attained through the use of bridge and amplifier circuits and a three-action control unit with recorder. The temperature sensor (one leg of a Wheatstone bridge) is a germanium resistor that has been repeatedly cycled from ambient to liquid helium temperatures to determine its stability and reproducibility. The unbalance of the bridge circuit is detected and fed into a servo-control unit, which possesses the controlling actions of proportional band, rate time and reset—the servo-unit's controlled current output is fed directly to the calibrator's heater coil. Current to the Wheatstone bridge circuit is supplied by a voltage divider circuit to permit a variation of current through the germanium sensor. This variable control is essential since, for sensitive temperature control the apparatus, the same currents cannot be applied at all temperatures. Because of the low specific heats and extremely high values of sensor resistance at low temperatures, too large a sensor current can cause: (a) excessive Joule heating of the sensor; and (b) a dissipation of heat (from the sensor into the calibrator) that is large compared with the heat output of the controlling heater coil. At the high calibration temperatures larger sensor currents are required, than at low temperatures, to obtain sufficient sensor sensitivity. In general, the temperature is controlled by the above method to about 0.0001 °K.

### 5.3. Resistance Measurements

Potential and current measurements are made on a double-bank six-dial potentiometer equipped with an external reversing switch. The advantage of the double-bank unit is that it relieves the necessity of repositioning dials when transferring from potential to current measurements. One bank of the potentiometer is used for measuring current. An external standard resistor is connected in series with the resistors to be calibrated and thus, from a measurement of

the potential across the standard resistor ( $I = \frac{E}{R}$ ), a value is obtained for the current through the total external circuit. The external reversing switch permits reversal of currents within the potentiometer and the external circuit, which includes the germanium thermometers. The measuring circuit is shown in figure 7. With this arrangement the thermal emf's may be detected and their effect on the measurements eliminated.

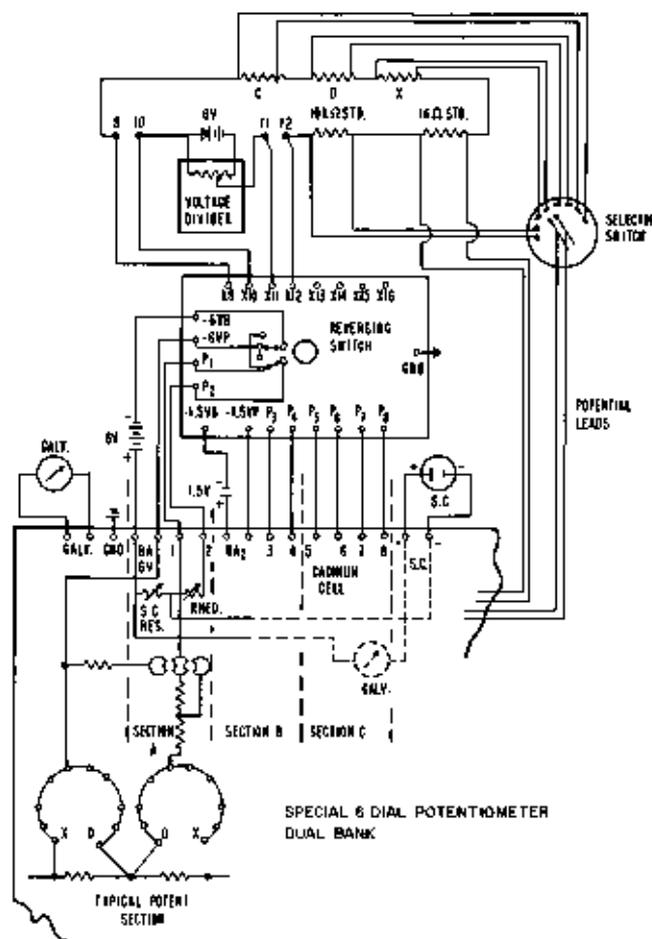


FIGURE 7. Schematic of the d-c resistance measurement circuit. This potentiometer actually consists of 3 potentiometer units in series (A, B, and C). Two sets of switches are connected to each decade to provide the "dual bank" feature.

### 5.4. Calibration Techniques

For many germanium thermometers, the resistor is a four-lead element, as shown in figure 6; two leads are for the current source and two for potential measurements. In the calibrator the current leads of the uncalibrated resistors are connected in series with the current leads of the calibrated thermometers to the current source at the terminal ring (E) of figure 5. The potential leads are wired individually to the platinum lugs which have electrical lead lines communicating to the potentiometer. The calibrated thermometers C and D are controlling parameters for determining the point-by-point resistance-temperature relation of the uncalibrated thermometers. Since an approximate temperature-resistance calibration of the sensor resistor is available, we are able to dial (for each calibration point) a particular value for this resistance on the Wheatstone bridge and then make final adjustments after comparing the two calibrated thermometer resistance-measurements with their previous values, i.e., temperature-resistance relation from the acoustical thermometer measurements. After the temperature constancy of the desired calibration point is clearly demonstrated, several series of potential measurements are made to determine the resistance of both the calibrated reference standards (C and D) and the submitted unknowns.

The complete calibration of submitted resistors is performed twice, covering the range 2 to 20 °K; in general, the temperature of a calibration point (column 1 of tables 3 through 5) must be within a few millidegrees of the calibration temperatures associated with resistors C and D (column 1 of tables 1 and 2); the differences of indicated temperatures between the standards C and D cannot exceed  $\pm 0.001$  °K; and, if possible, the two separate calibrations of the submitted resistors should be consistent within  $\pm 0.002$  °K.

TABLE 3. Resistor X

$T_{\text{data}}^a$	$R_{\text{data}}^a$	$R_{\text{ref}}^b$	$R_{\text{data}} - R_{\text{ref}}$ $\Delta R$	$\frac{dR}{dT}^c$ (Ohms/0.001 °K)	$\Delta T^d$ (0.001 °K)
°K	Ohms	Ohms	Ohms		
2.321	12.473	12.473.6	-1.0	-15.0	.1
2.805	7.584.2	7.580.8	3.4	-6.74	-0.5
3.207	5.458.2	5.462.5	-4.3	-4.5	1.1
4.206	2.944.5	2.940.5	3.8	-1.54	-2.5
5.020	1.991.5	1.994.6	-3.1	-0.87	3.5
6.060	1.307.5	1.306.0	1.5	-.50	-3.0
6.977	932.73	933.39	-0.66	-.33	2.0
8.065	647.37	647.17	.20	-.21	-1.0
8.989	486.50	486.32	-.18	-.14	-.1
9.869	376.44	376.40	.04	-1.03	-4
10.905	289.57	289.26	.31	-.071	-1.6
12.018	223.35	223.44	-.09	-.049	1.3
12.963	183.57	183.64	-.07	-.036	1.9
14.036	150.42	150.41	.01	-.026	-0.4
14.994	128.21	128.18	.03	-.020	-1.5
16.050	109.55	109.53	.02	-.016	-1.3
16.970	96.422	96.376	.046	-.013	-3.5
18.004	84.619	84.557	-.038	-.010	3.8
18.938	75.967	75.901	-.034	-.0085	1.7
20.048	67.490				

<sup>a</sup>  $T_{\text{data}}$  and  $R_{\text{data}}$  refer to calibration quantities, temperature and resistance.  
<sup>b</sup>  $R_{\text{ref}}$  is the resistance, at the temperature  $T_{\text{data}}$ , obtained by generating resistance as a function of temperature from the evaluated polynomial coefficients  $A_n$ .  
<sup>c</sup>  $\frac{dR}{dT}$  is generated from the evaluated coefficients  $A_n$ .  
<sup>d</sup>  $\Delta T$  is obtained by dividing  $(R_{\text{data}} - R_{\text{ref}})$  by  $\frac{dR}{dT}$ .

TABLE 4. Resistor Y

$T_{\text{data}}^a$	$R_{\text{data}}^a$	$R_{\text{ref}}^b$	$R_{\text{data}} - R_{\text{ref}}$ $\Delta R$	$\frac{dR}{dT}^c$ (Ohms/0.001 °K)	$\Delta T^d$ (0.001 °K)
°K	Ohms	Ohms	Ohms		
2.322	71.352	71.441	-89.0	-180	0.5
2.807	22.038	21.946	92.0	-50	-1.8
3.208	9.273	9.311.3	-38	-18.2	-2.1
4.208	1.921.0	1.919.5	1.5	-2.4	-0.6
5.019	826.04	828.27	-2.23	-0.71	3.1
6.060	400.48	398.17	2.31	-.23	-10.0
6.976	253.51	253.44	0.07	-.107	-0.7
8.065	169.44	169.70	-.26	-.034	4.8
8.990	129.30	129.70	-.40	-.034	11.8
9.867	103.81	103.98	-.17	-.024	7.0
10.901	83.627	83.623	.004	-.017	-0.2
12.017	67.719	67.607	.112	-.012	-9.3
12.962	57.482	57.380	.104	-.0096	-10.8
14.048	48.274	48.201	.073	-.0075	-9.7
14.993	41.852	41.820	.032	-.0061	-5.2
16.051	35.977	35.996	-.019	-.0050	3.8
16.969	31.781	31.816	-.035	-.0042	8.3
18.006	27.805	27.859	-.054	-.0035	15.4
18.940	24.820	24.852	-.032	-.0030	10.7
20.049	21.880	21.831	.049	-.0025	-19.6

<sup>a</sup>  $T_{\text{data}}$  and  $R_{\text{data}}$  refer to calibration quantities, temperature and resistance.  
<sup>b</sup>  $R_{\text{ref}}$  is the resistance, at the temperature  $T_{\text{data}}$ , obtained by generating resistance as a function of temperature from the evaluated polynomial coefficients  $A_n$ .  
<sup>c</sup>  $\frac{dR}{dT}$  is generated from the evaluated coefficients  $A_n$ .  
<sup>d</sup>  $\Delta T$  is obtained by dividing  $(R_{\text{data}} - R_{\text{ref}})$  by  $\frac{dR}{dT}$ .

TABLE 5. Resistor Z

$T_{\text{data}}^a$	$R_{\text{data}}^a$	$R_{\text{ref}}^b$	$R_{\text{data}} - R_{\text{ref}}$ $\Delta R$	$\frac{dR}{dT}^c$ (Ohms/0.001 °K)	$\Delta T^d$ (0.001 °K)
°K	Ohms	Ohms	Ohms		
2.319	1,918.8	1,918.9	-0.1	-1.45	0.07
2.806	1,404.7	1,404.5	.2	-0.76	-.3
3.208	1,156.5	1,156.6	-.1	-.50	.6
4.203	829.33	829.54	-.21	-.31	-1.4
5.018	695.22	695.42	-.20	-.20	1.5
6.059	596.69	596.58	.11	-.071	-1.5
6.977	543.81	543.93	-.12	-.046	2.6
8.068	504.17	504.17	.00	-.029	0.0
8.988	482.13	482.07	.06	-.020	-3.0
9.889	466.52	466.45	.07	-.015	-4.7
10.901	452.99	452.97	.02	-.012	-1.7
12.018	440.23	440.30	-.07	-.011	6.4
12.962	429.69	429.78	-.09	-.011	8.2
14.048	416.56	416.59	-.03	-.013	2.3
14.993	403.63	403.60	.03	-.015	-2.0
16.049	387.11	387.05	.06	-.017	-3.5
16.970	370.98	370.89	.09	-.018	-5.0
18.004	351.02	351.08	-.06	-.020	3.0
18.940	331.84	331.96	-.12	-.021	5.7
20.048	308.51	308.45	.06	-.021	-2.9

<sup>a</sup>  $T_{\text{data}}$  and  $R_{\text{data}}$  refer to calibration quantities, temperature and resistance.  
<sup>b</sup>  $R_{\text{ref}}$  is the resistance, at the temperature  $T_{\text{data}}$ , obtained by generating resistance as a function of temperature from the evaluated polynomial coefficients  $A_n$ .  
<sup>c</sup>  $\frac{dR}{dT}$  is generated from the evaluated coefficients  $A_n$ .  
<sup>d</sup>  $\Delta T$  is obtained by dividing  $(R_{\text{data}} - R_{\text{ref}})$  by  $\frac{dR}{dT}$ .

Periodically during calibrations, when the extent of Joule heating in a submitted resistor is not known, power tests are performed. This is to insure that the resistor current is sufficiently low that Joule heating effects are well below 1 mdg.

Certain features of the calibration apparatus merit mentioning. A high vacuum is maintained within the inner can; the resistors are mounted (within the copper core) in a stopcock grease medium; the region of the copper core, which contains the spiralled leads, has been covered with a suitable varnish to enhance thermal equilibrium between the core and leads; the heater coil has been varnished onto its containing form; and the current leads from each resistor are thermally anchored to the copper core. These features aid in

promoting thermal equilibrium (between the germanium resistors) to such an extent that a thermal gradient has not been detected—even though the heater has been operated at powers that have varied by an order of magnitude. (The power variance occurs, during measurements at a constant temperature, as the vacuum in the outer can [figure 3] is allowed to change.)

## 6. Results and Conclusions

The calibration data for three resistors that have been calibrated against resistor C and D are presented in figure 8 and in columns 1 and 2 of tables 3, 4, and 5. The resistors are from three different commercial sources and may, perhaps, be considered typical of available resistors. The resistors are labeled as X, Y, and Z and their calibration data have been treated on the computer in a manner paralleling that previously described in this paper for resistors C and D. We have attempted to fit the data for each resistor to a polynomial of the form

$$\log_{10} R = \sum_{n=0}^m A_n (\log_{10} T)^n$$

Resistors X and Z appeared to be reasonably represented by an eighth order polynomial; for Y, the significance of the polynomial coefficients became doubtful for the seventh and eighth orders. Generations for X, Y, and Z, based upon 9, 7, and 9 coefficients, respectively, were performed to obtain the quantities

$$R, \frac{dR}{dT}, \frac{d^2R}{dT^2} \text{ and } \frac{1}{R} \frac{dR}{dT}$$

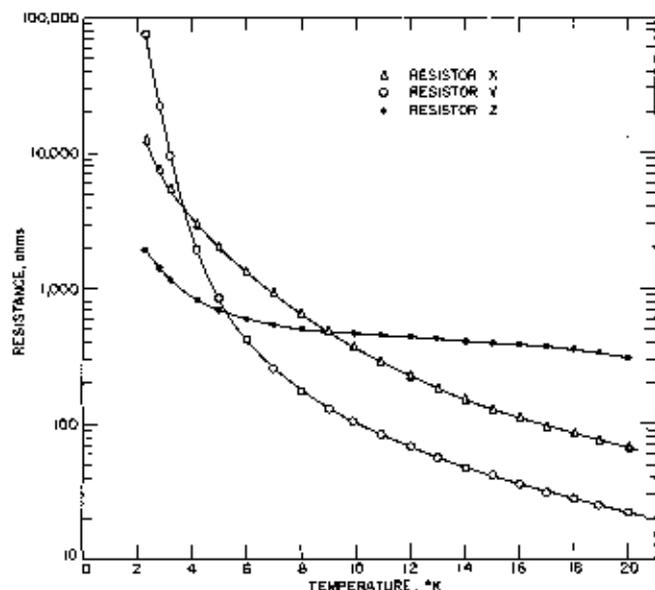


FIGURE 8. Plot of resistance as a function of temperature:  $\Delta$ , resistor X;  $\circ$ , resistor Y; and  $\bullet$ , resistor Z.

as a function of temperature. Since the columns of tables 3, 4, and 5 correspond to those of tables 1 and 2, columns 6 indicate how well the calibrations of resistors X, Y, and Z are represented by the fitted polynomials. For these resistors also, elimination of the data at the lowest temperature will generally result in a more accurate functional fit at lower polynomial orders.

It is to be noted, for resistor Z in figure 8 that the  $R$ - $T$  relationship is not characterized by a monotonically decreasing derivative  $\frac{dR}{dT}$ . Indeed the generation of the second derivative  $\frac{d^2R}{dT^2}$  clearly exhibits a change in sign between 12.060 and 12.070 °K, and also between 20.480 and 20.490 °K. (The change in sign is not significant at the higher temperatures, since 20.480 °K exceeds the range of the calibration data.) Such a condition indicates that caution must be exercised in attempting to use our functional relationship for interpolating temperatures and resistances that are intermediate to the calibration points of resistor Z. Perhaps a better approach for interpolation would result from functionally fitting selected segments of the calibration data.

To repeat, columns 6 of tables 3, 4, and 5 demonstrate that the functional representations for resistors X and Z are more accurate than is indicated for resistor Y. While this can possibly be partially attributed to Y's low values of  $\frac{dR}{dT}$  at the highest temperatures, one must note that the polynomial fit for resistor Y covers a much larger range of resistance than is the case for resistors X and Z. However, it is probably more significant to note that Y is basically a two lead resistor while X and Z involve four lead contacts. As has been described earlier in this paper, the electrical measurements of resistance are made by a direct-current, potentiometric technique. In the case of resistors X and Z, current connections to the germanium crystal are distinctly separate from the potential

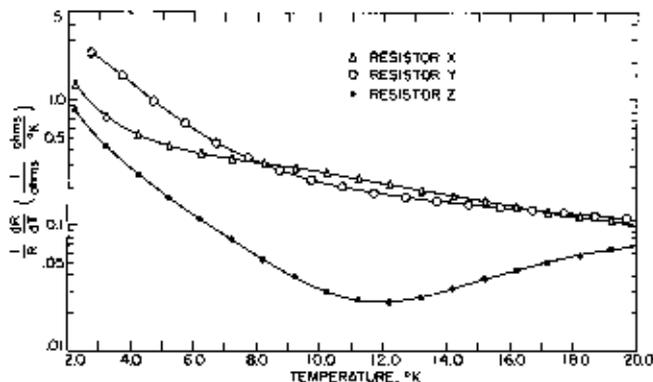


FIGURE 9. Resistance-temperature sensitivity,  $\frac{1}{R} \frac{dR}{dT} \left( \frac{1}{^\circ K} \right)$ , as a function of temperature ( $^\circ K$ ):  $\Delta$ , resistor X;  $\circ$ , resistor Y; and  $\bullet$ , resistor Z.

leads. For resistor Y, a current and potential lead are connected to each of the two leads that lead to the germanium resistor. It is possible that lead or contact resistances introduce additional uncertainties in the determination of Y's resistance.

Figure 9 indicates resistor sensitivities. The sensitivity is the fractional change of resistance per °K as a function of the absolute temperature. Quite obviously, depending upon the user's requirements, selection of a particular type of resistor is important since the sensitivities,  $\frac{1}{R} \frac{dR}{dT}$ , as well as the absolute magnitude of resistance,  $R$ , are variable—depending upon the concentration of impurities that have been added to the germanium.

Calibration certificates for submitted resistors are issued for calibrations at approximately the temperatures indicated in column 1 of table 1 or table 2; it is expected that these values of temperature are reproduced within  $\pm 0.002$  °K; and the measuring current at each temperature is specified. For the user's information we attempt to fit the calibration data with a polynomial and include the results as part of the calibration service.

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